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16. ABSTRACT

The importance of Quality Assurance is discussed. The requirements of a quality assurance program are divided into three functions; namely, the Control of Quality, the Determination of Quality, and the Documentation of Quality, The Control of Quality is defined as the regulation of factors that influence the reliability of the data. The Determination of Quality is defined as the use of procedures to estimate how good are the final results obtained in terms of quanitative accuracy and precision and qualitative accuracy. The documentation of Quality is defined as the use of procedures to form a systematic, intelligible, complete and permanent record of the control of quality and determination of quality.

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ELEMENTS OF A LABORATORY QUALITY ASSURANCE PROGRAM

by

Ronald C. Dressman

A quality assurance (QA) program benefits a business or other institutions engaged in analytical work in many ways. These ways include such management related benefits as the improvement and maintenance of morale and professionalism; stimulated productivity; greater control over operating costs; an increase in the marketability of the service; and overall suitability of operations. These benefits are no less important to the water utility than to any other organization providing a consumer product.

As rewarding as these benefits may be, however, none has the essential character that describes the real reason for establishing a laboratory quality assurance (LQA) program; namely, to ensure valid analytical results. For that reason alone, laboratory quality assurance is indispensible to the analytical laboratory. A laboratory that does not practice quality assurance turns out meaningless results; meaningless because the degree of validity of those results is an unknown quantity. When the degree of validity of results, whether qualitative or quantitative, is unknown, those results can not be intelligently used in a decision making process. Thus, the user of the data has the responsibility to demand that results be validated. Ensuring the validity of analytical results, however, is primarily the responsibility of those people actually engaged in performing or supervising the analytical operations.

The program outlined in the following discussion focuses on this responsibility in the analytical laboratory, and is appropriate whether that laboratory is engaged in routine analytical measurements or research, and applies for purposes of safe drinking water, whether that laboratory

is a part of the utility, itself, or is a state, federal, commercial or university laboratory providing service to the utility.

Many factors affect the quality of analytical results, and many degrees of control can be exercised over these factors. Certainly all laboratories exercise at least some of these controls and believe in the validity of their own results. Often overlooked, however, is the need to establish, in a formal way, a measure of confidence that the degree of validity has been correctly stated. The only way to establish confidence that laboratory results possess a known degree of validity is through the use of a highly visible and sound laboratory quality assurance program that protects against errors going undetected and uncorrected.

An effective way for laboratory personnel and their supervisors to meet the requirements of quality assurance for actual analytical laboratory operations is to adopt a program such as the one described here that establishes standard operating procedures (SOPs) for analysis and for the three functions of laboratory quality assurance; namely, the control of quality, the determination of quality, and the documentation of quality.

Standard operating procedures for analysis may be defined as validated methods documented as step-by-step procedures that may be applied by any experienced analyst, without variation, to produce repeatable and reproducible results. These SOPs have the specific details of QA common to the normal operating procedure for a given method of analysis incorporated into the text of that analytical method, or appended to it as a LQA supplement (although a well written analytical method will have little need of being supplemented). This LQA program also requires that a deviation from the SOP for any specific work assignment be documented in writing in the appropriate notebook or file so that the data later can be judged accordingly.

Because many specific requirements of quality assurance may vary for any or all of the following reasons; namely, the method of analyses, the designs of a project or the ultimate use of the data, SOPs of a more general nature that provide basic guidance for all laboratory operations are provided for each of the three LQA functions in addition to the SOPs for analyses. Many of the elements that comprise each of the three functions of LQA are generally applicable to any laboratory no matter what type of analyses are being performed. The elements are presented below, and some are briefly discussed.

I. Standard Operating Procedure for the Control of Quality

Individual operating procedures in a laboratory that regulate the factors that influence the reliability of the data are the elements that make up the SOP for the control of quality. These procedures may or may not be directly involved in the analytical process, but they all exercise a control somewhere in the midst of the overall operation of the laboratory. These factors are often taken for granted in day-to-day operations and so frequently are denied the careful attention they deserve. The factors themselves are listed as main headings and are accompanied by a description of the regulating elements.

1. Sample Handling Requirements

a. Planning the Analysis

Planning the analysis is often overlooked as an item of quality control because quality control is most often thought of in terms of activity at the bench. Planning the analysis in the context of this discussion can mean anything from designing a research project or planning a routine sampling operation within your own facility,

to approving requests for the analysis of samples originating outside your laboratory. In any case, certain information is essential to the proper conduct of analysis that, if not provided in the planning stage, usually results in information of a lesser or less well known quality than if such information were provided. The following list of items, not necessarily in any particular order of importance, should always be considered:

Requesting Authority

Estimate of Participation, i.e., at what interval and over what period of time will samples be submitted

Project Title and Description

Type of Analyses

Number of Samples (including number of replicates)

Sample Identification Code

Sample Description

Any Other Information That Could Influence the Conduct of the Analysis; e.g., likely chemical interferences that may have to be dealt with in an exceptional way.

b. Sampling

Sampling is often considered the responsiblity of someone outside of the laboratory and, as a consequence, is often not considered as it relates to the strict requirements of the analysis. Certain details, however, are often the responsibility of the analytical laboratory and must be carefully controlled to avoid adversely affecting the reliability of the data. The analytical laboratory must

° Provide or require sampling containers that have been cleaned according to the requirements of the method of analysis (see glassware).

- Provide or require containers that satisfy method requirements for sample size, replication of results and preservation of the chemical integrity of the sample.
- Provide or require chemical preservatives to satisfy the requirements of the method of analysis and the use of the data.

c. Sample Receiving

Employ a controlled, systemtatic procedure of receiving samples, whether such samples are being generated within the laboratory or from without, to ensure the integrity of the samples. The integrity of samples can be seriously compromised if samples when received are not stored in a proper and timely manner, and when samples are analyzed in an untimely manner because a record of either or both time of sampling and time of arrival has not been kept.

2. Glassware

a) Treat glassware to ensure the removal of all traces of the parameter(s) of interest and contaminants that could interfere with the analysis. The treatment itself must not introduce interference with a specific analysis.

3. Materials, Reagents, Solvents and Gases, etc.

a) Carefully select each item to conform to the specifications defined in the method of analysis.

b) Test each new discreet supply of these items to verify their performance capabilities, verify their freedom from impurities that would interfere with the analysis, and to quantify the level of background each produces.

c) Properly store these items to protect them from degradation and contamination, and make note of the expected shelf life.

d) Periodically retest any stored item whose stability may be suspect, or that may be susceptible to becoming contaminated.

e) Analyze method blanks to measure the bias that these items collectively have on analytical results. Combine all items that are a part of the analytical method into the analysis of a sample of known zero or near zero value for the parameter(s) of interest.

f) Analyze method blank as frequently as is necessary to reliably establish the bias.

4. Chemical Solutions

a) Before preparing solutions of chemicals, be certain (I.7.a) that all measuring devices such as volumetric glassware, syringes, analytical balances, meters etc. are in properly calibrated working order, and capable of measuring to the degree of accuracy required by the analytical method.

b) Verify the concentration of solute in Standard Solution against a primary analytical reference standard if available, or by the most direct, reliable, approved method of measurement when such standards are not available.

c) To minimize the effect of the error associated with aliquot measurement, take aliquots for dilution in volumes as large as is practical.

d) When preparing a series of standard solutions for precision testing, prepare, whenever possible, one large volume with a single dose and then aliquot that solution for testing, rather than dose each test solution individually.

e) Label all solutions with the following information:

Solute(s)	Concentration	Preparer
Solvent(s)	Date Prepared	Source of Solute

f) Carefully store solutions of chemicals to protect them from degradation and contamination.

g) Periodically retest any stored solution whose stability may be suspect, or that may be susceptible to becoming contaminated.

5. Analytical Methodology

a) Use a method that is validated for, and meets the requirements for, selectivity, accuracy, precision and range.

c) If a validated method is unavailable from any source, whatever method-development, modification or evaluation is undertaken must be performed in the context of a quality assurance plan that concludes with at least an informal method validation study (B.1.a).

6. Training the Analyst

a) Provide formal classroom training for the analyst in preparation for assignments requiring specialized knowledge of instrumentation and applications.

b) Provide on-the-job training under close supervision by a qualified instructor.

c) Place special emphasis on training the analyst in the importance of quality control and quality assurance.

d) Place special emphasis on the importance of technique and adherence to SOPs.

7. Auxiliary Equipment Calibration and Maintenance

a) Periodically check the calibration of auxiliary equipment with which measurements are made that in any way influence the outcome of analytical operations. These include pH meters, pyrometers, balances, gauges, thermometers etc.

b) Perform necessary maintenance to this equipment on a regularly scheduled basis.

8. Analytical Instrument Calibration and Maintenance

a) When first installed, and on a regular basis thereafter, perform internal instrument calibration checks as instructed in the instrument manual.

b) Analyze analytical reference standards as qualitiative and quantitative checks on instrument performance during the course of analyzing samples.

c) Establish performance criteria for judging when data from instrument performance checks signal the need to make adjustments in the way the instrument is operating.

d) Perform routine preventive maintenance to the instrument on a regularly scheduled basis.

e) Develop trouble shooting skills that will enable you to recognize problems, their cause, and the appropriate corrective action quickly and accurately, and thus avoid more complicated failures and dependence upon outside servicing agencies.

f) Employ servicing contracts whenever they may provide an overall advantage to laboratory operations.

9. Facilities and Services

a) Establish the control of the laboratory environment to eliminate sample contaminating influences, to optimize instrument operating conditions; e.g., temperature and humidity and to promote efficiency and maintain safe working conditions.

b) Ensure that services such as electricity, water, air, gas and vacuum meet specifications for efficient and reliable performance.

10. Intermediate Samples

a) To assist in identifying the point of breakdown in method performance, devise stepwise performance-evaluation tests that provide quality assurance for intermediate samples, analagous to that provided for the overall method. Intermediate Samples are defined as being samples (e.g., eluates, extracts, concentrates) produced by isolation and concentration procedures.

11. Measurements

a) At all times operate within the linear range of the instrument; make dilutions to samples as necessary.

b) Monitor instrument sensitivity at regular intervals on day of analysis for the parameters of interest

c) Redefine the limit of detection for each parameter whenever there is a change in sensitivity.

d) Where the analytical instrument itself does not provide an accurate quantification of the parameter of interest being detected, quantify the response in one of the following ways:

- use a calibration curve constructed from several replicate results for three levels of response to the parameter of interest, the extremes of which bracket the expected level of response during sample analysis;
- use either a single result or the average for several replicate results for standards responding within a range of response defined by the QA incorporated into the method SOP for that same parameter in the sample, to do ratio and proportioning;

° use response factors calculated from either internal or external control-compound data acquired under identical conditions of analysis at response levels within a range of response defined by the QA incorporated into the method SOP for sought after parameters in the sample.

e) When measurements must be made manually from strip-chart or other graphic recordings of response, make enough replicate measurements to provide reasonable assurance of accuracy.

f) Double-check all calculations and manual measurements of graphically recorded instrument response

g) Abide by rules for significant figures

h) Whenever possible, apply statistics to the evaluation of raw data assembled to determine whether a given operation is in control or out of control.

II. Standard Operating Procedures for the Determination of Quality

Individual operating procedures in a laboratory that estimate how good the final results are in terms of quantitative accuracy and precision, and qualitiative accuracy, are the elements that make up the SOP for the determination of quality. These procedures are deliberately planned, formal procedures specifically aimed at defining the validity of a result. Generally, several alternatives are available to perform this function, many of which are discussed here in brief terms. Three categories of control are dedicated to quantitative results and two to qualitative results.

- Quantitative Results

1. Method-Validation and Performance-Monitoring - External Control

a) Informally validate new methods by analyzing analytical reference samples prepared as quantitative blinds (concentration(s) unknown to the analyst of parameter(s) of interest).

b) Formally validate new methods by participating in round robin studies involving analytical reference samples prepared as quantitative blinds.

c) Analyze analytical reference samples at least once every six months on each instrument and whenever changes are made in the analytical system that could affect the known quality of results. Analytical reference samples may be obtained from the Environmental Monitoring and Support Laboratory (EMSL) of EPA, Cincinnati, or any other source capable of providing the necessary blind samples, and should be analyzed by a certified analyst (see II.2.b).

2. Analysts Certification and Performance-Monitoring - External Control

a) Certify new analysts on validated methods by providing analytical reference samples prepared as quantitative blinds.

b) Repeat the blind sample analysis at least once every six months for each analyst. The 6-month analyst certification and 6-month validation serve a reciprocal function for that analyst and that instrument.

3. Method Performance-Monitoring - Internal Control

a) Determine the accuracy of data being generated by analyzing samples (d,e) fortified with a known quantity of the parameter(s) of interest, or a compound or compounds representative of the parameter(s) of interest.

b) Determine the precision of the data being generated by analyzing replicates of the samples (d,e) fortified with a known quantity of the parameter(s) of interest, or a compound or compounds representative of the parameter(s) of interest.

c) Determine the precision of the data being generated by analyzing replicates of the true samples.

d) Fortify reagent water (i.e., Super-Q or equivalent) to isolate the effects of the method on data quality when determining accuracy and precision (a,b).

e) Fortify the actual sample matrix, after establishing the background concentration of the parameter(s) of interest, to determine the effect of the sample matrix on data quality when determining accuracy and precision (a,b).

f) As an alternative to fortifying independent samples (II.3.a,b,c), fortify each true sample with an internal control compound to determine the quality of results directly on each sample analysis.

g) Treat fortified samples to every step of the method; include sample handling; i.e., sampling procedure, shipping, preservation techniques and storage as necessary. Determine the need to treat fortified samples to sample handling procedures by independent study, if possible, otherwise monitor these effects throughout the course of the work.

h) Set the frequency with which fortified samples and true-sample replicates are run, select the sample matrix for fortification and the levels of concentration, and choose the type of control used and the limits of acceptable accuracy and precision, based on the following factors:

Parameter(s) of Interest	Requirements of the Method
Use of the Data	Analytical Complexity
Sample Type	Volume of Work
Knowledge of Sample Source	Signals from Control Data

Statistics to Be Used for Determining Precision and Accuracy

i) Determine the quality of data derived from "extended" analytical samples - i.e., samples requiring further treatment by procedures (e.g., clean-up) neither usually required to produce an analytical sample nor to which the scheduled, method quality assurance applies - by preparing simulated analytical samples and carrying them through the extended procedure. Carry original control sample through the extended procedure whenever possible. Apply standard operating procedures for quality assurance to all aspects of the extended procedure.

j) Relate the quality of true-sample result to the most recently acquired piece of control data or averaged block of control data if control charts are not feasible.

k) Relate the quality of true-sample results to control-sample data in a continuing way through the use of quality control charts if the following conditions are satisfied:

- The number of analyses on a given instrument by a given analyst is sufficiently continuous; i.e., the continuity of said analyses is not broken by either or both frequent or prolonged periods of inactivity that invalidate the purpose of the control chart, which is to be able to detect trends toward being out of control.
- The method of analysis is sufficiently redundant; ie., whatever affected any true-sample or control-sample result can reasonably be expected to have affected any other result obtained in the time frame of control. (Solvent extractions for organic chemicals are not sufficiently redundant, for

example, and are best controlled by the use of the internal control of each sample. Automated analysis for Cl^- on the other hand is sufficiently redundant.)

- Qualitative Results

1. Method Performance-Monitoring, and Analyst Certification and Performance-Monitoring - External Control

   a) Monitor the ability to make correct individual compound or species identifications, by substituting analytical-reference samples prepared as qualitative blinds (identification(s) as well as concentration(s) unknown to the analyst of parameter(s) of interest) for those prepared as quantitative blinds (II.1.c; II.2.a,b).

2. Confirmation

   a) If necessary, confirm the identification of individual compounds or species by one of the following procedures:

      1. Significantly vary the conditions of either or both isolation and detection.

      2. Analyze a replicate sample by a uniquely different method of analysis.

III. Standard Operating Procedure for the Documentation of Quality

Individual operating procedures in a laboratory that form a systematic, intelligible, complete and permanent record of the control of quality and the determination of quality make up the SOP for the documentation of quality. These procedures should make it possible for anyone not having first hand knowledge of the laboratory operation to trace the handling of a sample from the time of request for analysis to the reporting of results, and to observe the degree of validity of those results simply by reviewing laboratory records.

Nothing, of course, is magical about documentation. Our SOP for the documentation of quality describes procedures specific to our laboratory and deals with such elements as Files; Logs; Notebooks; Chain-of-Custody Records; Reports; the preservation of computer printouts, charts, graphics; the storage of data and records of audits. Some of the more generally applicable procedures for some of those elements of documentation are included here.

1. Files

   A central file on each work assignment should be established to either contain all information and related documents for that assignment or contain information locating same.

2. Master Sample Log

   The master sample log is the first step in ensuring the sample receives proper treatment, and is the first step in chain-of-custody proceedings should they be required. The following information is entered on each log sheet:

   Date Received and by whom (signature);

   Source;

   Description of Samples to include:

   Indenfication, type, size, how many, number of replicates, preservative used, shipping temperature, date sampled;

   Description of Samples to include either or both:

   How and where stored, by whom (signature) and date;

   To whom transferred (signature) and date;

   Date Analyzed.

3. Sample "Back" Log

   Each staff member in charge of analyzing for a particular parameter must keep a sample "Back" Log that is a record of samples in

storage to be analyzed under his or her responsibility. This sample "Back" Log must be updated at the time the staff member takes custody of the samples, and is retained in the appropriate file. The pertinent information may simply be copied from the Master Log onto individual "Back" Log sheets.

4. Chain-of-Custody Record

   a) When "strict" chain-of-custody procedures such as for enforcement samples apply, refer to EMSL-CI/1005 of October 1, 1980.

   b) For work not requiring "strict" chain-of-custody procedures, the traceability of sample handling is documented by the use of the Master Log, which traces the sample from the time it arrives in the laboratory until it is transferred into the hands of the staff member in charge of the analysis for immediate analysis or storage; and by the use of the Notebooks (C.8.), which trace the handling of the sample from the moment it is taken for analysis to the time the analysis is completed; and by the official report form, which traces the communication of results.

5. Analytical-Instrument-System Activity Logs

   a) Attach to each analytical-instrument system a log book to record the performance of calibration and maintenance procedures, incidents of troubleshooting, and occurrence of service calls.

   b) Date and describe each occurrence of maintenance.

   c) Date each occurrence of trouble and describe the symptoms, the problem, the solution (including results of testing) and recommendations.

   d) Date and describe each occurrence of service requiring a service call.

6. Auxiliary-Equipment Activity Log

   a) In the designated laboratory location keep a logbook of records of maintenance, troubleshooting and service to auxiliary equipment such as ovens, balances, meters, etc.

7. Materials, Reagents & Chemical-Solutions Activity Log

   a) In the designated laboratory location keep a log book of records of tests performed on materials, reagents, and chemical solutions to determine their suitability for use, to calibrate them, or to monitor their stability. Date each test and enter the name of the analyst. Also in the log, record a reference or a description of each SOP used for each test.

8. Notebooks (Routine Analysis)

   a) Use numbered notebooks with numbered pages.

   b) At the beginning of each assignment, identify the work assignment and give the sample series or number of samples. Date the entry.

   c) Reference the Standard Operating Procedures (SOPs) for method of analysis (including specific conditions of analysis where choices are permitted) and requirements of quality assurance for each technical assistance assignment. Date the entry.

   d) When SOPs are not available for reference, or deviations from SOPs are exercised, record the operating procedures in the notebook. Date the entry.

   e) Identify by sample number and record raw sample data for each true and control sample in chronological order as it is produced. Raw data is defined as any numerical value that is obtained by test as a part of the method of analysis and that influences the final results. Date each day's work and record the identity of the instrument and the analyst (signature).

f) In the same notebook containing the raw data, and associated with the corresponding raw data, record the final result as the value to be reported after reconciliation with control data and conversion to appropriate reporting units.

9. Notebooks (Research Projects)

a) In addition to the above notebook procedures for routine analyses (III.8.), record all observations, discussions and conclusions throughout the progress of the project.

10. Computer Printouts

a) Computer printouts containing either or both raw data and final results may be substituted for notebook entries. Any information not contained on the printout but requiring documentation must be entered into a lab notebook (III.8). Printouts must be assigned an identification (ID) number and must be referenced in the notebook by that number.

b) Label printouts with the ID number, the notebook number and page number, and file in the designated laboratory location.

11. Charts

a) When quality-control charts are used, display them as readily visual, documentary evidence of the state of analytical quality during the entire course of a unique laboratory operation.

b) Label quality-control charts and any other charts or graphs with an ID number, the notebook number and page number, and file in the designated laboratory location. Charts etc. must be referenced in relevant fashion in the notebook by their ID number.

12. Graphics

a) Graphic displays such as chromatograms and spectragrams are preserved based on the need to provide visual evidence to support the use of the data.

b) Label each graphic or set of graphics with the conditions of analysis, with an ID number, the notebook number and page number, and file in the designated laboratory location. Graphics must be referenced in the notebook in relevant fashion by the ID number.

13. Reports

a) For data generated for routine analysis:

° At the end of each assignment, report by memo, letter or official report-form the following information, and file a copy of each such transmittal in the designated laboratory location:

Project Identification

Sample Identification

Parameters Sought & Their Limits of Detection

Final Results

Estimation of Precision and Accuracy

Type of Correction Applied to Raw Data

Reference to Operating Procedure for Analytical Method

Reference to Operating Procedure for Quality Assurrance

Discussion of the Reliability of the Data

Interpretation of the Data if Possible

b) For data generated by in-house research projects:

- File data summaries and copies of progress reports, including final report, in designated laboratory location.

14. Data Banks

a) Assure that final results of true-sample analyses are in proper units and supply required information by memo, letter or official report form to the appropriate information-storage office if one exists. Retain a copy of each such transmittal in the designated laboratory location.

15. Audits

a) File records of formal internal and external quality-assurance audits in the designated laboratory location.

b) Include in the audit file, records of Method Validations, Analyst Certifications, Inspection Reports and Reports of Corrective Action.

SUMMARY

The purpose of laboratory quality assurance is to ensure, assess and document that all data collected, stored, reported, or used are scientifically valid. Ensuring the validity of analytical results is primarily the responsibility of the people actually engaged in performing or supervising the analytical operations. An effective way of meeting the requirements of quality assurance is to adopt a program that establishes standard operating procedures for analysis and for the three functions of quality assurance, which are the control of quality, the determination of quality and the documentation of quality.